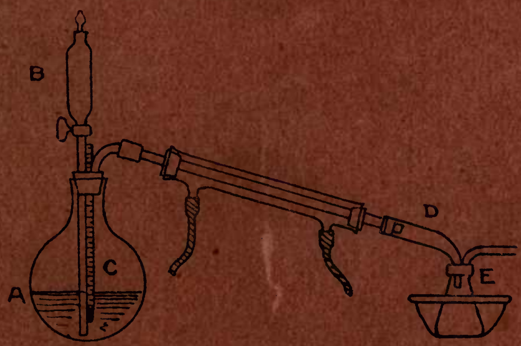
A
LABORATORY MANUAL

W. R. ORNDORFF



D. C. HEATH & CO.

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A
LABORATORY MANUAL

CONTAINING

DIRECTIONS FOR A COURSE OF EXPERIMENTS
IN ORGANIC CHEMISTRY

SYSTEMATICALLY ARRANGED TO ACCOMPANY
REMSEN'S ORGANIC CHEMISTRY

BY

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PREFACE.

At the author's request I have carefully examined this Manual; and, as a result, I have recommended its publication, in the belief that it will be a valuable adjunct to my "Introduction to the Study of the Compounds of Carbon." Great care has evidently been taken to determine the best conditions for each experiment; and, in many cases, the directions given are undoubtedly better than those given in my book. This is largely due to the fact that the author has, for a number of years, had charge of the work of preparation of compounds of carbon in a large laboratory; and he has therefore had an excellent opportunity thoroughly to test different methods, — an opportunity which he has well utilized.

IRA REMSEN.

Baltimore, Md., March 6, 1893.

EXPERIMENT 1.

FRACTIONAL DISTILLATION.

Fit up an apparatus like the one shown in Fig. 1 (text-book, page 5), using a 1-liter distilling-flask (Fig. 2, text-book), in place of the flask and thermometer tube. Add 300 c.c. of alcohol and 300 c.c. of water to the flask, and separate the two by fractional distillation, following closely the directions given on pages 6 and 7 of the text-book. Distil *slowly* and *regularly*. Measure each fraction, and the residue left in the flask at the end of each series of distillations. Fill in the blanks in the following table, and draw your own conclusions from the results.

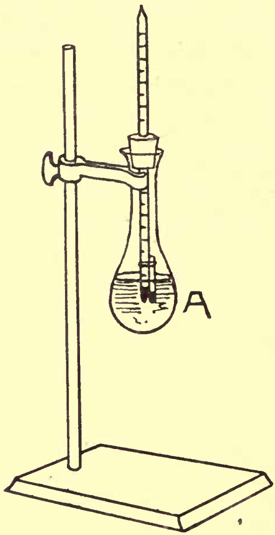
FRACTIONS.	DISTILLATIONS.				
	1.	2.	3.	4.	5.
78°– 83°					
83°– 88°					
88°– 93°					
93°– 98°					
98°–100°					
Residues					
Totals					

For further information regarding all kinds of distillation, and for the methods of determining boiling-points, the student is referred to Lassar-Cohn's very valuable book, "Arbeitsmethoden für Organisch-Chemische Laboratorien" (Voss, Leipzig, 1891).

EXPERIMENT 2.

DETERMINATION OF MELTING-POINTS.

Determine the melting-points of the substances given below, proceeding as directed in the text-book, page 9, using, however, the apparatus here figured: *A* is a pear-shaped flask, of 100 c.c. capacity, $\frac{3}{4}$ filled with *colorless* concentrated sulphuric acid. The thermometer is held in place by a loosely fitting cork with a groove filed in it. The capillary tube is fastened to the thermometer by a rubber band, or, better, by a small piece of platinum wire, so that the substance and the bulb of the thermometer are side by side. By fastening two capillary tubes, one on each side of the thermometer, two determinations may be made at once. Should the sulphuric acid become dark colored (from the organic matter suspended in it), it may be rendered colorless by removing the thermometer, adding a small quantity of concentrated nitric acid, and heating until the nitric acid has been driven off. (Hood.) For further details



concerning melting-point determinations, see Lassar-Cohn's book, page 75.

SUBSTANCES.	MELTING-POINTS.	
	Observed.	Given in Text.
Naphthalene.		
Urea.		
Succinic acid.		
Anthracene.		
Anthraquinone.		

EXPERIMENT 3.

MARSH GAS.

Grind together intimately 20 grams of *fused* sodium acetate and 40 grams of powdered soda-lime. Heat the mixture in an iron or copper retort with a triple burner. Collect the gas evolved over water in wide-mouthed cylinders of 200 c.c. capacity. What is left in the retort? Prove it. Write out the reaction.

Determine the properties of the gas, including color, odor, specific gravity (lighter or heavier than air), inflammability, solubility, etc. Mix one volume of marsh gas and two volumes of oxygen and explode by applying a lighted taper. Why are the gases mixed in this proportion? What are the products of the explosion?

CAUTION.—*In working with gases see that all joints of the apparatus are tight before beginning the experiment.*

EXPERIMENT 4.

CHLOROFORM.

275 grams of bleaching-powder and 800 c.c. of water are put into a 3-liter balloon-flask, and thoroughly mixed. The flask is closed with a 3-hole rubber stopper. Through one hole passes a separating funnel, reaching to the bottom of the flask; through the second, a tube bent at right angles, and also reaching to the bottom of the flask; and through the third, the exit tube. Connect the flask with the condenser and receiver, heat on a water-bath, and add gradually, through the separating funnel, a mixture of 22 grams of acetone and 70 c.c. of water, shaking the flask constantly during the addition of the acetone and water. After all the acetone has been added and the reaction is completed, steam is passed in through the tube reaching to the bottom of the flask, and the distillation in steam is continued as long as chloroform distils with the water.

Separate the chloroform from the water by means of a separating funnel; wash it with dilute caustic soda solution (?), and then with water (?). Remove the water completely, add an equal volume of concentrated sulphuric acid (?), and shake together in a separating funnel. Drain off *completely* the sulphuric acid and distil the chloroform, noting the boiling-point.

Save a specimen of the chloroform, and determine its properties, — color, odor, taste, boiling-point, specific gravity (lighter or heavier than water), solubility, inflammability.

What is formed when chloroform is heated with an alcoholic solution of caustic potash?

Write out reactions after acetone and chloral have been studied.

EXPERIMENT 5.[1]

IODOFORM.

1.

Dissolve 15 grams of potassium carbonate in 100 c.c. of water, filter the solution if necessary, and add 16 grams of alcohol (94%). The flask containing the solution is warmed to 70°C, and 20 grams of iodine are then *gradually* added, with constant shaking of the flask. A gas is given off; what is it? Filter off the iodoform, wash with water, dry in the air on filter-paper, and recrystallize from alcohol. Determine its melting-point, solubility in water, alcohol, ether, carbon bisulphide; crystal form, taste, odor, and color. The filtrate from the iodoform is evaporated to crystallization and the crystals examined. What are they? Prove it.

Save a specimen of the crystallized iodoform and the potassium iodide.

Reactions are to be written out after chloral has been considered.

[1] Make iodoform by either of the two methods here given. The second has given the better results in this laboratory.

EXPERIMENT 5.

IODOFORM.

2.

Dissolve 25 grams of potassium iodide in 500 c.c. of water and add 5 grams of acetone. To this mixture add, through a dropping-funnel or burette, with constant shaking, a dilute solution of sodium hypochlorite as long as a precipitate is formed. Allow the precipitate to settle, decant off the liquid, wash with water two or three times, filter, drain thoroughly, dry on filter-paper, and recrystallize from alcohol. Does any iodide remain in solution? Determine the melting-point, solubility in water, alcohol, ether, carbon bisulphide, etc.; crystal form, taste, odor, and color.

Save a specimen of the crystallized iodoform. Write out the reactions after chloral and acetone have been considered.

The solution of sodium hypochlorite used in this experiment may be readily made by precipitating all the calcium in a solution of bleaching-powder (chloride of lime) with a solution of sodium carbonate. A slight excess of sodium carbonate will not interfere with the reaction.

EXPERIMENT 6.[1]

ETHYL BROMIDE.

(Hood.)

1.

10 grams of *dry* (?) amorphous phosphorus and 60 grams of absolute (?) alcohol are brought together in a 500 c.c. round-bottomed flask which is kept cold by ice-water; 60 grams (19 c.c.) of bromine are then gradually added through a separating-funnel, drop by drop, with constant shaking of the flask. After all the bromine has been added, the flask is connected with a return condenser (see Fig. 8, page 70, text-book) and gently heated on a water-bath until the reaction is completed. The condenser is then turned down, and the ethyl bromide distilled off. Wash this with *dilute* caustic soda solution (?), then with water; drain off the water, and dry by warming *slightly* with fused calcium chloride, using a return condenser (?). Pour off from the calcium chloride into a dry distilling-flask and distil, noting the boiling-point.

Determine color, odor, taste, specific gravity (heavier or lighter than water), inflammability (Hood). Save specimen of the pure product. What

[1] Either of the two methods given may be used to prepare ethyl bromide; the second has given better results in this laboratory.

EXPERIMENT 6.[1]

ETHYL BROMIDE.

(HOOD.)

1.

10 grams of *dry* (?) amorphous phosphorus and 60 grams of absolute (?) alcohol are brought together in a 500 c.c. round-bottomed flask which is kept cold by ice-water; 60 grams (19 c.c.) of bromine are then gradually added through a separating-funnel, drop by drop, with constant shaking of the flask. After all the bromine has been added, the flask is connected with a return condenser (see Fig. 8, page 70, text-book) and gently heated on a water-bath until the reaction is completed. The condenser is then turned down, and the ethyl bromide distilled off. Wash this with *dilute* caustic soda solution (?), then with water; drain off the water, and dry by warming *slightly* with fused calcium chloride, using a return condenser (?). Pour off from the calcium chloride into a dry distilling-flask and distil, noting the boiling-point.

Determine color, odor, taste, specific gravity (heavier or lighter than water), inflammability (Hood). Save specimen of the pure product. What

[1] Either of the two methods given may be used to prepare ethyl bromide; the second has given better results in this laboratory.

is left in the flask in which the ethyl bromide was made? Prove it. Add a few drops of ethyl bromide to some silver nitrate solution and heat. Explain what takes place. Write out all reactions.

EXPERIMENT 6.

ETHYL BROMIDE.

(Hood.)

2.

100 grams of finely powdered potassium bromide are placed in a 250 c.c. distilling-flask, and a *cooled* mixture of 42 grams of absolute alcohol (96% will do) and 83 grams of concentrated sulphuric acid is slowly added. The flask is then connected with the condenser and heated on a sand-bath until all the ethyl bromide has been distilled. The ethyl bromide is then separated from the water, washed first with dilute caustic soda solution, then with water, and dried by warming on a water-bath with fused calcium chloride (using a return condenser — see Fig. 8, page 70, text-book). The product is then distilled, carefully noting the boiling-point. Save a specimen of the pure ethyl bromide.

Determine its physical properties: color, taste, odor, specific gravity (lighter or heavier than water), inflammability (Hood), boiling-point, etc. Determine the action of silver nitrate solution at boiling heat on ethyl bromide. What remains in the flask in which the ethyl bromide was made? Prove it. Write out all reactions.

EXPERIMENT 7.

FERMENTATION OF GLUCOSE.

Dissolve 150 grams of glucose in about $1\frac{1}{2}$ liters of water, add 60 c.c. of a solution of Pasteur salts.[1] Put the mixture into a 2-liter flask, add $\frac{1}{4}$ cake of compressed yeast (cut fine), and warm to 25° C. on a water-bath. Connect the flask with a cylinder containing limewater (protected from the air by a layer of kerosene or benzene). Keep the temperature *as near* 25° as possible (by warming on a water-bath), until fermentation ceases; then allow the yeast to settle, and draw off the clear liquid. Distil off 500 c.c. of this, using a Hempel distilling-tube (?), and test the distillate for alcohol (see Roscoe and Schorlemmer's Treatise on Chemistry, Vol. III. Part I. page 318). Write out all reactions.

Explain why Pasteur salts are used and why the limewater must be protected from the air. What gas comes off? Will the alcohol burn? Try it.

For further information on the subject of fermentation the student is referred to Schutzenberger's book on Fermentation.

[1] The formula for Pasteur salts, based on an analysis of the inorganic constituents of the yeast plant, is as follows: —

Potassium phosphate	2.00 pts.
Calcium "	0.20 "
Magnesium sulphate	0.20 "
Ammonium tartrate	10.00 "
Water	857.60 "

EXPERIMENT 8.

ABSOLUTE ALCOHOL.

Heat, on the water-bath, 1 liter of alcohol (95 %) with 500 grams of *quicklime*, broken into small lumps (not powder), in a 3-liter short-necked balloon flask provided with a return condenser (see text-book, page 70, Fig. 8). After boiling the alcohol with the lime for two hours, distil off the alcohol, and determine whether it is free from water (see Roscoe and Schorlemmer's Treatise on Chemistry, Vol. III. Part I. page 298; and Beilstein's "Organische Chemie," Vol. I. page 237). If not, repeat the process with the distillate.

Explain the process, and determine the specific gravity of the alcohol and the per cent of water it contains.

Determine the properties of pure alcohol, including color, odor, taste, boiling-point, inflammability. Does its vapor mixed with air explode? Try it. Does it solidify when cooled? What use is made of this fact? Is alcohol a good solvent? Try it.

The student is recommended to read the chapter on alcoholometry and the methods of determining the amount of alcohol in wines, beer, etc., in Roscoe and Schorlemmer's Treatise on Chemistry, Vol. III. Part I. page 301.

EXPERIMENT 9.

CALCIUM ETHYL SULPHATE.

Add 98 grams of concentrated sulphuric acid to 138 grams of absolute alcohol without cooling, and heat the mixture on the water or steam bath for a quarter of an hour. After the mixture is *cold*, pour it slowly, and with constant stirring, into a porcelain dish containing crushed ice or snow (?). Then dilute with ice-water to about a liter and a half. The acid is now *nearly* neutralized by adding slaked lime or precipitated chalk, ice being added from time to time to prevent any rise of temperature (?). The solution is then filtered off from the calcium sulphate through a muslin filter stretched on a wooden frame, or, better, by the method of reverse filtration. The apparatus for this latter process consists of a small funnel which is covered with white cotton cloth, and which is connected by a rubber tube with a bottle, the bottle in turn being connected with a suction-pump. The funnel is placed in the mixture to be filtered, and the pump started. The liquid is drawn through the funnel into the bottle, and is quickly and completely filtered. The precipitate is then treated with $\frac{1}{2}$ liter of *cold* water and the liquid again filtered off. Clear limewater is now added to the combined filtrates to alkaline reaction (?), and this solution is evaporated to crystallization on the steam or water-bath, taking care to filter off the calcium

sulphate which separates out as the solution is concentrated. Explain. After the first crop of crystals has formed, filter them off, using a Witt plate and a filtering-flask connected with a pump, and repeat the process with the mother liquor. Drain the crystals thoroughly and dry on drying-paper.

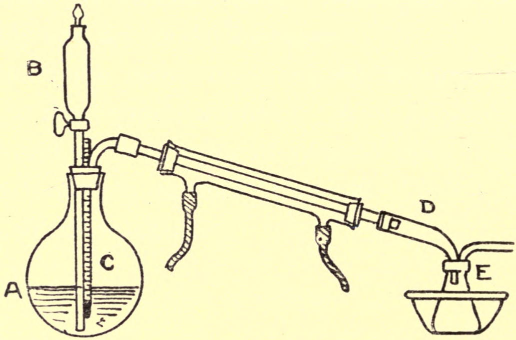
Determine the color, taste, crystal form, solubility, etc., of the crystals obtained. Save a specimen. Does the salt contain water of crystallization? Try it. Does it contain carbon? Prove it.

Write out all reactions.

EXPERIMENT 10.

ETHER.

Arrange an apparatus like the one shown in the figure below.



A is a round-bottomed, wide-necked flask of 2 liters' capacity.

B is a cylindrical separating-funnel reaching to the bottom of *A*.

C, a short thermometer, the bulb of which *must* dip below the surface of the liquid.

D, an adapter which is connected with the receiver by a doubly bored stopper.

E, a receiver, which *must* be surrounded with a freezing mixture (ice and salt).

Into the flask *A* put a cooled mixture of 306 grams of concentrated sulphuric acid and 170 grams of alcohol (90%). Heat the mixture in the flask *A* until the temperature reaches 140° C. Then cautiously open the stopcock of the separating-funnel and let a

slow stream of alcohol *in the form of vapor* bubble through the liquid, regulating the flow of alcohol so that the temperature of the mixture is kept as nearly 140° C. as possible (not below 140° and not above 145°). When 500 grams of alcohol have been run in, the operation is stopped. The distillate consists of two layers, and contains, besides ether, water, alcohol, and sulphurous acid. The watery layer is removed by means of a liter separating-funnel, and the ether is washed first with dilute caustic soda solution (?), then two or three times with small quantities of distilled water (?). The washed ether is now treated with one-half its weight of fused calcium chloride, and distilled on a water-bath through a Hempel distilling-tube, taking care that the temperature does not rise above 50° C. The distillate *must* be kept cold by ice-water. Weigh the ether obtained and see how the yield accords with that required by theory.

Determine the boiling-point, specific gravity, color, solubility in alcohol and water, taste, and odor. Place a few drops on the hand and note the effect produced. Mix a *little* in a wide, strong cylinder with air, and apply a lighted taper. Explain. Is it a good solvent? Try it. Does water dissolve ether? Does ether dissolve water?

Write out all reactions. Save a specimen of ether.

CAUTION. — *In working with ether always avoid the neighborhood of flames* (?).

EXPERIMENT 11.

Put a few cubic centimeters of ether into a small evaporating-dish and put the dish on a sand-bath. Apply a flame. Note the result.

What are the products of the combustion? Prove it.

EXPERIMENT 12.

Into a thin glass test-tube put 5 or 10 c.c. of water and place the tube in a *small* beaker containing some ether. Through a glass tube, just reaching to the surface of the ether, pass air from a foot bellows or a blast. In a few moments the water in the test-tube will be frozen. Explain.

EXPERIMENT 13.

ALDEHYDE.

Dissolve 5 grams of potassium bichromate in water and add gradually 15 c.c. of concentrated sulphuric acid. Is there any change of color? What is formed? Now add a few cubic centimeters of alcohol and warm the tube. Notice the odor. What is formed? Does the solution change color? Explain. What has become of the potassium bichromate and the alcohol? What is left in the solution? Prove it and write out all reactions.

EXPERIMENT 14.

ALDEHYDE.

225 grams of potassium bichromate (in small pieces not powdered) are placed in a 3-liter round-bottomed flask, and the flask surrounded with a freezing mixture. Then add through a separating-funnel a cool mixture of 125 grams of alcohol (90%), 900 c.c. of water, and 300 grams of concentrated sulphuric acid, shaking the flask during the addition of the mixture, so as to avoid any great rise of temperature. The flask is then removed from the freezing mixture, connected with a Hempel distilling-tube (40 to 50 c.m. long), a condenser, and receiver, *the latter surrounded with a freezing mixture.* Heat the flask gently and collect the distillate, taking care that the temperature shown by the thermometer in the Hempel tube does not exceed 50° C. Distil until aldehyde ceases to come off.

Determine the taste, odor, color, boiling-point, solubility, etc., of the aldehyde obtained. Is it lighter or heavier than water? Does it dissolve iodine, sulphur, phosphorus? Try it. Heat a few drops of aldehyde with caustic potash solution. Note change of color and odor (characteristic of aldehyde). Save a few drops for Experiment 15.

Cool 10 c.c. of this aldehyde in a small flask in a freezing mixture and pass in a few bubbles of hydrochloric acid gas. What takes place? Explain. Fil-

ter off the crystals, wash them with alcohol, and cool the first filtrate in a mixture of concentrated hydrochloric acid and crushed ice (or ice and crystallized calcium chloride). Note result. Explain. To 10 c.c. of the aldehyde add 20 c.c. of anhydrous ether, cool in a freezing mixture, and saturate with dry ammonia gas, obtained by heating a concentrated solution of aqueous ammonia and passing the gas evolved through a cylinder filled with quicklime or soda-lime (?). Note result. Explain. Filter off the crystals, wash with ether, and dry on drying paper. Let the filtrate evaporate spontaneously in a crystallyzing-dish, when more crystals will be obtained.

Save specimens of aldehyde, metaldehyde, paraldehyde, and aldehyde ammonia.

Write out all reactions.

EXPERIMENT 15.

DETECTION OF ALDEHYDE.

Take 10 c.c. of a solution of silver nitrate, add cautiously drop by drop a *dilute* solution of ammonia until the silver oxide first precipitated is *just* dissolved, taking care to avoid *any* excess of ammonia. An ammoniacal solution of silver oxide is thus obtained.

Clean a test-tube thoroughly by boiling some concentrated nitric acid in it, pouring out the acid and washing with distilled water. Heat the ammoniacal silver oxide solution to boiling in the cleaned test-tube, then add a drop of the aqueous solution of the aldehyde obtained in Experiment 14, noting the result. Explain. What becomes of the aldehyde? What use is made of this method in the arts?

Test the delicacy of this reaction both for aldehyde and for silver. (See in this connection Roscoe and Schorlemmer's Treatise on Chemistry, Vol. III. page 478.)

Write out all reactions and save a specimen of the silver mirror.

EXPERIMENT 16.

FORMIC ACID.

Into a 500 c.c. dry distilling-flask put 200 grams of crystallized oxalic acid and 200 grams of *anhydrous* glycerin (if the glycerin is not anhydrous, heat it in a porcelain dish under the hood to 175° C. for an hour or two). Insert a thermometer through the cork, so that the bulb dips below the surface of the glycerin, and connect the flask with a condenser and a receiver. Heat gently with a burner, keeping the temperature of the liquid between 100°–115° C. What gas is given off? When this gas ceases to be evolved, and after the contents of the flask have cooled to 75°, add 50 grams more of the crystallized oxalic acid, and heat as before. Repeat this addition of oxalic acid until 250 c.c. of distillate have collected in the receiver. Put the glycerin into the bottle marked "Glycerin Residues," and set aside 25 c.c. of the distillate for the experiments given below. Divide the rest into three portions. Neutralize one portion with chalk or slaked lime, filter, evaporate to crystallization, and save specimen of the calcium salt. Warm the second portion with a slight excess of lead carbonate or oxide, decant through a filter, and extract the precipitate two or three times with boiling water (?). Evaporate the combined filtrates to crystallization and save a specimen of the lead salt. Heat the third portion until saturated with freshly precipitated copper hydroxide

(obtained by precipitating a solution of copper sulphate with caustic soda solution and washing the precipitate *thoroughly* with water), filter, evaporate the filtrate to crystallization, and save a specimen of the copper salt.

With the 25 c.c. of the distillate perform the following experiments: —

(*a*) Heat a small quantity with some mercuric oxide and note the result. Explain.

(*b*) Heat some with a solution of silver nitrate and describe and explain what takes place.

(Explain why these two reactions are characteristic of formic acid.)

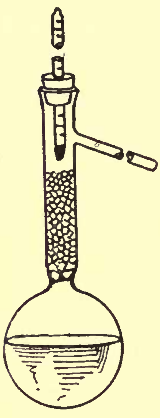
Describe both the physical and chemical properties of all the salts made; save specimens of all and of the distillate. Write out all reactions.

(Use the material in the bottle marked "Glycerin Residues" to start with, if it is available, instead of glycerin itself, and add only 50 grams of oxalic acid at a time.)

EXPERIMENT 17.

ACETIC ACID.

150 grams of *fused* sodium acetate are powdered and put into a 500 c.c. dry distilling-flask. The flask is cooled, and 180 grams of concentrated sulphuric acid (sp. gr. 1.842) are gradually added through a funnel-tube reaching to the bottom of the flask. The flask is then connected with a condenser and receiver and heated on a sand-bath. The distillate is subjected to fractional distillation, using a short Hempel distilling-tube, or, better, a distilling-flask,[1] with a column of glass beads in the neck (see figure).



First an aqueous acid passes over and is collected separately, but between 117° and 119° the anhydrous acid distils. Cool this distillate (between 117°–119°) with ice-water, and note what takes place. Save specimen of the glacial acid. Why is the first distillate not anhydrous?

A portion of the first distillate of the aqueous acid is *carefully* neutralized with caustic soda solution and divided into four portions. One portion is heated to boiling, and then a slight excess of mer-

[1] These flasks, which are exceedingly convenient for fractional distillation, may be procured from Gerhardt, or from Eimer & Amend.

curous nitrate solution added. Explain what takes place. The second portion is also heated to boiling, and a tolerably concentrated solution of silver nitrate added. Note what takes place. Explain. The third portion is evaporated to dryness, on a steam or water bath, with some powdered arsenic trioxide, and a *little* of the resulting mass heated in a test-tube when the characteristic odor of cacodyl oxide is given off. Explain. To the fourth portion add a little concentrated sulphuric acid and alcohol, and heat. Notice the odor. Explain.

Write out all reactions, and show how you could distinguish between formic and acetic acids.

Describe the properties of acetic acid, and give some of the methods by which this substance may be detected in solution.

EXPERIMENT 18.

Make up a solution containing ferric chloride, aluminum, manganese, zinc, cobalt, and nickel chlorides. Nearly neutralize any free acid present with a solution of sodium carbonate (?). If a precipitate is formed dissolve in a small quantity of acetic acid. Add enough of a concentrated solution of sodium acetate to convert all the metals present into acetates (taking care to *avoid* a large excess of the reagent), and boil the solution. Note what takes place. What is volatilized? Explain. Filter off the precipitate and test the filtrate for iron, aluminum, and the other metals. Draw your own conclusions from the results, and show how these facts could be utilized in analytical chemistry. Could you separate ferrous iron from ferric by this method? Explain.

EXPERIMENT 19.

To a mixture of equal parts of acetic acid and alcohol in a test-tube add some concentrated sulphuric acid, and heat. Note what takes place. To what is the pleasant and characteristic odor due? What part does the sulphuric acid play?

Write out reactions, and determine the delicacy of this reaction for the detection of small quantities of acetic acid and salts of acetic acid.

EXPERIMENT 20.

ACETYL CHLORIDE.

(Hood.)

43 grams of glacial acetic acid are put into a *dry* (?) 250 c.c. round-bottomed flask, the flask cooled with ice-water, and 65 grams of phosphorus trichloride (weigh this under the hood (?)) are gradually added, with constant shaking. Connect the flask with a return condenser, and heat it on a water-bath to 50°–60° C. until almost all the hydrochloric acid gas has been expelled. The condenser is then turned down and the acetyl chloride distilled off, care being taken to protect the distillate from the action of the moisture in the air by means of a tube containing calcium chloride. The distillate is then placed in a small, dry distilling-flask, and redistilled with the same precautions as before.

What remains in the flask in which the acetyl chloride was made? Prove it. Save this material and put into proper bottle.

Determine all the properties of acetyl chloride, including color, odor, boiling-point, action on moist air. Add a few drops to some water in a test-tube and note what takes place. Explain. Put aside some of the chloride for Experiment 21. Save specimen in a small sealed tube and write out all reactions.

EXPERIMENT 21.

Treat a few cubic centimeters of absolute alcohol with acetyl chloride and note what takes place. What gas is given off? What is formed? Explain. Repeat the experiment, using water instead of alcohol. Explain. What conclusions would you draw regarding the relation between water and alcohol from this experiment? What use is made of acetyl chloride in the laboratory? Write out all reactions.

EXPERIMENT 22.

ETHYL ACETATE.

(*Acetic Ether.*)

Arrange an apparatus like that used in the preparation of ether (see Experiment 10, page 16). Into the flask *A* put 50 grams of concentrated sulphuric acid and 50 grams of absolute alcohol (93% will do). Heat till the temperature reaches 130°–140° C., and then add gradually through the separating-funnel a mixture of equal volumes of glacial acetic acid and absolute alcohol (93% will do).

Separate the crude acetic ether from the water and wash with caustic soda solution; dehydrate in the usual manner with fused calcium chloride, and subject to fractional distillation.

Determine color, taste, odor, boiling-point, specific gravity, solubility, inflammability, etc. Is there any analogy in the method of preparation of this acetic ether and the method used to make ordinary ether? What else is formed here besides acetic ether? Save specimen. Write all reactions.

EXPERIMENT 23.

SAPONIFICATION.

Arrange an apparatus as shown in Fig. 8, page 70, text-book, using a round-bottomed 500 c.c. flask. Calculate the amount of solid caustic potash necessary to decompose 20 grams of the ethyl acetate made in Experiment 22. Dissolve this amount plus 5 grams' excess in 200 c.c. of water, add the 20 grams of ethyl acetate, and boil together on a sand-bath until the odor of ethyl acetate has disappeared. Then distil off 100 c.c. of the liquid and examine the distillate. What does it contain?

Let the mixture in the flask cool, and *when cold* acidify with dilute sulphuric acid and again distil. What passes over now? Prove it. Write out all reactions.

Given a mixture of ethyl acetate and ethyl alcohol, show how you could determine the amount of ethyl acetate present.

EXPERIMENT 24.[1]

POTASSIUM CYANIDE.

(HOOD.)

100 grams of potassium ferrocyanide are dehydrated by heating the powdered salt in a porcelain dish in an air-bath to 110° C. until it ceases to lose weight. The dehydrated salt is powdered again if necessary, and then heated in an iron or copper retort provided with a cover and outlet-tube (such as was used for making marsh gas in Experiment 3), to a red heat with a triple burner or in a Fletcher gas-furnace. A gas comes off. Collect some over water and determine what it is. Continue heating the retort until gas ceases to come off, excluding air from the retort by keeping the outlet-tube under water. Then remove the outlet-tube from the water and let the retort cool. Take out the contents of the retort, grind in a mortar, and extract once or twice with boiling 50% alcohol. Filter and evaporate to crystallization. Determine solubility and crystal form of the salt. Does it resemble potassium chloride in any way? Has it any odor? Explain. Is the salt deliquescent? Does the aqueous solution decompose? Try it. What are the products? Is the aqueous solution alkaline? Explain. Does an aqueous solution of the salt dissolve the cyanides of mercury, gold, etc.? Try it. What

[1] Experiments 24, 26, and 27 are to be postponed until after urea has been considered.

use is made of this property? Does the fused salt reduce metallic oxides? Try it with some lead oxide (PbO) in a test-tube. Is the salt poisonous? Save a small specimen and keep the rest for use in Experiment 27. Write out all reactions.

EXPERIMENT 25.[1]

(Hood.)

POTASSIUM FERRICYANIDE.

1.

Dissolve 50 grams of the crystallized potassium ferrocyanide in 150 c.c. of luke warm water in a 250 c.c. flask. Filter the solution if necessary. Calculate the amount of bromine required to convert the 50 grams of ferrocyanide into ferricyanide. Weigh off this amount of bromine under the hood, and add it *gradually* (by means of a separating-funnel, having its lower end drawn out into a capillary tube), to the solution of ferrocyanide, shaking constantly while the bromine is being added. The solution is then evaporated to one-half its volume, and the ferricyanide allowed to crystallize out. The mother liquor, on being evaporated down, also yields a further crop of crystals. Save a specimen of the salt, and determine color, taste, crystal form, solubility, etc., of the potassium ferricyanide. Does the solution decompose in the light? Try it. What use is made of this fact? Is it a good oxidizing agent? Try it in alkaline solution with some lead oxide. Does potassium ferrocyanide contain water of crystallization? Does the ferricyanide? To a small quantity of

[1] Either method may be used in making potassium ferricyanide. The second has given better results, however, in this laboratory.

a saturated solution of the ferrocyanide in a test-tube add an equal volume of fuming hydrochloric acid. Add two or three volumes of fuming hydrochloric acid to one volume of a saturated solution of the ferricyanide. Explain what takes place in each case. Write out all reactions.

EXPERIMENT 25.

POTASSIUM FERRICYANIDE.

2.

Dissolve 50 grams of crystallized potassium ferrocyanide in 300 c.c. of water and add 10 grams of concentrated hydrochloric acid. Then run in from a burette a cold filtered solution of bleaching-powder until the solution no longer gives a precipitate of prussian blue with ferric chloride solution (testing the solution from time to time by taking out a drop or two). Neutralize any excess of hydrochloric acid present with precipitated chalk, filter the solution, and evaporate to crystallization. The first crop of crystals will be pure; the second may contain a small quantity of calcium chloride.

Save a specimen of the salt, and examine it as directed in Experiment 25, 1.

EXPERIMENT 26.

POTASSIUM CYANATE.

Mix intimately 4 parts of *dehydrated* powdered potassium ferrocyanide with 3 parts of dry powdered potassium bichromate. A *little* of this mixture is placed in a porcelain or, better, an iron dish and heated (considerably below redness) until the mixture becomes like tinder and blackens. The rest of the mixture is then added, little by little, each quantity being allowed to blacken before the next is added (complete oxidation of the cyanide to cyanate is thus effected). After cooling, the contents of the dish are extracted several times with boiling alcohol (80%). Filter, and cool the alcoholic solution to 0°. Filter off the crystals formed, using a Witt plate and suction-pump. Dry on drying-paper. Continue the extraction until the black mass is exhausted. Yield, 42% of the ferrocyanide used. Make 30 grams of the cyanate, and calculate the amounts of ferrocyanide and bichromate necessary. Save a small specimen of the salt, and put away 20 grams of the cyanate for Experiment 48. Determine crystal form, solubility, color, taste, odor, etc. Does the aqueous solution decompose on standing? Try it. What are the products? Add some dilute sulphuric acid to a solution of the cyanate, and explain what takes place. Is there any resemblance between potassium

cyanate and potassium hypochlorite? Show this by the method of formation, one from cyanogen and the other from chlorine and a solution of potassium hydroxide. Write out all reactions.

EXPERIMENT 27.

POTASSIUM SULPHOCYANATE.

An aqueous solution of potassium cyanide (65 parts of the dry salt) is heated with flowers of sulphur (32 parts) until all the sulphur is dissolved. Filter the solution and evaporate to cystallization. Drain off the crystals and repeat the evaporation with the mother liquor. Use 20 grams of the potassium cyanide made in Experiment 24 and calculate the amount of sulphur necessary to form the sulphocyanate.

Determine properties of the salt, including color, taste, odor, crystal form, solubility, melting-point, etc. What takes place when the crystals are left exposed to the air? Is there any analogy between the formation of the cyanate (Experiment 26) and the sulphocyanate? What use is made of the sulphocyanate in the laboratory? Heat some of the salt in a porcelain crucible and note changes in color. Let cool and note any changes. Make some mercuric sulphocyanate, dry, roll into small pellets, and ignite (Pharaoh's serpents). Explain.

Save a specimen of the potassium salt and write out all reactions.

EXPERIMENT 28.

Dissolve 5 grams of potassium sulphocyanate in water, and note any change in the temperature of the water. Explain.

EXPERIMENT 29.

AMMONIUM SULPHOCYANATE.

Mix 60 grams of alcohol (95%), 80 grams of ammonia solution (sp. gr. 0.912), and 35 to 40 grams of carbon bisulphide, and allow to stand in a tightly stoppered bottle for one or more days until all the carbon bisulphide has dissolved. The process may be hastened by shaking the bottle from time to time. When all the carbon bisulphide has dissolved, evaporate the solution at a gentle heat on a steam or water bath (Hood) to one-third of the original volume. What goes off? Filter off any sulphur that may separate out while the solution is hot. On cooling, ammonium sulphocyanate crystallizes out. Filter off the crystals and repeat the evaporation with the mother liquor.

Determine crystal form, solubility, color, taste, odor, and melting-point of the salt.

Save a specimen of the salt, and write out all reactions.

EXPERIMENT 30.

PHENYL ISOCYANIDE.

(See text-book, page 90.)

Dissolve a small quantity of solid caustic potash in alcohol in a test-tube and add one or two drops of aniline. Then add a drop of chloroform to 100 c.c. of distilled water in a small flask, and shake the mixture thoroughly. Now add a drop or two of this water saturated with chloroform to the caustic potash solution containing the aniline, and heat the test-tube *under the hood.* The odor is that of phenyl isocyanide.

This reaction is a very delicate one, and may be used to detect the presence of any primary amine or of chloroform. Is aniline an amine? Write the reaction. What is the object of the caustic potash?

EXPERIMENT 31.

FLASHING-POINT OF KEROSENE.

Make an apparatus like the one figured on page 111 of the text-book, but of the following dimensions: *A* is a glass cylinder 35 m.m. wide and 175 m.m. high, and has a mark 60 m.m. from the bottom, and another at 70 m.m. from the bottom. The kerosene is poured in up to the lower mark. The cylinder is placed in a water-bath (a beaker filled with water up to the 60 m.m. mark on the cylinder), the temperature of which is raised slowly (one degree in two or three minutes). At each degree air is passed through for five seconds so rapidly that the foam on the kerosene reaches the second mark. At the same time a small flame is held at the mouth of the vessel *A*. When the vapor ignites and a bluish flame runs down to the surface of the oil, note the temperature. The first determination gives only approximate results. The kerosene is replaced by a *fresh* supply, and the observations are begun at a temperature a little below that obtained in the first determination. The temperature of the kerosene and that of the water-bath should differ only by one degree. Determinations should agree to within $\frac{1}{4}°$. The proportions given for the cylinder and the directions *must* be observed to get good results. Determine the flashing-point of several specimens of kerosene, and particularly of that kerosene you burn at your room. See if it is above the flashing-point required by law.

EXPERIMENT 32.

ALDEHYDE.

Mix equal weights (10 grams) of dry calcium formate and dry calcium acetate. Distil from an iron or copper retort. Collect some of the distillate in water. What does the water contain? Prove it. Write out the reaction.

EXPERIMENT 33.

PALMITIC AND STEARIC ACIDS.

Dissolve 10 grams of solid caustic potash in 150 c.c. of alcohol in a 250 c.c. flask and filter. Melt in a porcelain dish on a water-bath 10 grams of lard and then add the alcoholic solution of caustic potash. Evaporate to syrupy consistency slowly on the water-bath, with constant stirring. Convince yourself that the substance left is soap (?).

Dissolve the syrupy mass in a small quantity of *cold* water, and filter if necessary. Add to a little of this soap solution a few drops of a solution of (1) calcium sulphate, (2) magnesium sulphate, and (3) calcium acid carbonate, and explain what takes place in *each* case. What is meant by the terms permanent and temporary hardness of water? How do you determine the hardness of a water? How could you make a hard water soft?

Acidify the soap solution remaining with dilute sulphuric acid. A precipitate is formed. What is it? Filter off the precipitate and drain it thoroughly. Recrystallize it from alcohol and determine its melting-point and properties. Has the substance acid properties? Prove it.

Neutralize the filtrate exactly with a solution of potassium carbonate or potassium hydroxide, evaporate to dryness on the water-bath, and extract with absolute alcohol. Filter the alcoholic solution, and evaporate off the alcohol on the water or steam bath. What is the syrup which remains? Prove it.

Save specimens of the acids and the syrup. Write out all reactions after glycerol has been considered.

EXPERIMENT 34.

OXALIC ACID.

(Hood.)

In a long-necked flask, of about 1-liter capacity, heat gently 25 grams of cane sugar and 125 grams of nitric acid (sp. gr. 1.245). Gases are evolved. Explain. After the reaction is over, evaporate the solution to crystallization and let cool. Filter or pour off the mother liquor and add to it a smaller quantity of nitric acid, heat, and again evaporate to crystallization. Filter off the crystals, drain completely on a Witt plate, and recrystallize from distilled water.

Determine color, taste (poison), odor, solubility, crystal form, etc., of the acid. Is it a strong acid? Try it. Does it contain water of crystallization? Prove it. Does it sublime when heated? Try it. Is it a reducing agent? Why? Heat some of the acid in a small flask under the hood with concentrated sulphuric acid and prove that both oxides of carbon are set free. What does the acid yield when heated with glycerol? What is potassium tetroxalate? What use is made of this salt in the laboratory?

Save a specimen of oxalic acid. Write out reactions after glucose has been considered.

EXPERIMENT 35.

Dissolve some oxalic acid in water and add some dilute sulphuric acid (?). Now run in a little of a dilute solution of potassium permanganate and note what takes place. What becomes of the permanganate and of the oxalic acid? Prove it. Write out the reaction and calculate how much permanganate would be necessary to oxidize 5 grams of *crystallized* oxalic acid.

EXPERIMENT 36.

BASIC FERRIC SUCCINATE.

Dissolve about a gram of succinic acid in water, and neutralize *carefully* with dilute ammonia solution. Add some of this solution of ammonium succinate to a neutral solution of manganese chloride and ferric chloride, and boil. A precipitate is formed. What is it? Filter it off, and test the filtrate for iron and for manganese, and draw your own conclusions from the results. Has the filtrate an acid reaction? Explain. Write out all reactions. Compare results with those obtained in Experiment 18, page 26.

EXPERIMENT 37.

GLYCEROL.

Determine color, taste, odor, solubility, etc., of glycerol. Is it hygroscopic? Can it be distilled under ordinary pressure? What is its boiling-point? Does glycerol dissolve caustic potash, lead oxide (PbO), or calcium oxide? Explain.

Put 20 c.c. of glycerol into a 250 c.c. distilling-flask and distil with superheated steam. Does the glycerol distil with the steam? Prove it — see Roscoe and Schorlemmer's Treatise on Chemistry, Vol. III. Part II. page 350, for a method of detecting glycerol.

Compare reactions of the glycerol obtained in Experiment 33 with the ordinary glycerol. Are the two identical? Explain the formation of formic acid by the decomposition of oxalic acid in glycerol by heat.

EXPERIMENT 38.

FEHLING'S SOLUTION.

Dissolve 3.5 grams of crystallized copper sulphate in 50 c.c. of water to which one drop of sulphuric acid has been added. Bottle and label "Copper Sulphate Solution." Take 17.5 grams Rochelle salt (potassium sodium tartrate) and dissolve in 35 c.c. of water. Filter and add a solution of 5 grams of caustic soda in 10 c.c. of water. Make the solution up to 50 c.c., bottle and label "Alkaline Tartrate Solution." For use, mix equal parts of these solutions.

Into a small porcelain dish put 5 c.c. of each of the above solutions. Heat nearly to boiling and add a few drops of a one per cent solution of glucose. Note what takes place. Continue to add the glucose solution drop by drop until the precipitate settles and leaves the solution clear and colorless. Filter off the precipitate, wash with hot water, dry, and save specimen. What is this precipitate? What becomes of the glucose? Does a solution of glucose reduce an ammoniacal solution of silver oxide? Try it. What conclusions do you draw from these facts as to the constitution of glucose? Write out all reactions.

EXPERIMENT 40.

Dissolve 1.5 grams of cane sugar in 200 c.c. of distilled water. Test a little of this solution with the Fehling's solution, proceeding as directed in Experiment 38. Does any reduction of the copper solution take place? Now add to the sugar solution about ten drops of hydrochloric acid (sp. gr. 1.11), and heat the mixture on the water-bath to 100° for half an hour. Neutralize the solution exactly with a dilute solution of sodium carbonate, and again test it with Fehling's solution. Does any reduction take place now? What is the function of the hydrochloric acid? Explain.

Given a solution containing both cane sugar and glucose, show how you could estimate both by means of Fehling's solution.

EXPERIMENT 41.

CELLULOSE.

Treat some absorbent cotton with *dilute* caustic soda solution and warm gently; then wash with water, and repeat the process, using dilute hydrochloric acid. Again wash with water, then boil with alcohol once or twice, pour off the alcohol, and digest with ether. Remove the ether and dry. This treatment removes incrusting substances and leaves a comparatively pure cellulose. Has cellulose any crystalline form? Is it soluble in any of the ordinary solvents? Try it. Dissolve some pure cellulose (Swedish filter-paper) in a small quantity of concentrated sulphuric acid; dilute with much water, and boil the solution for half an hour. Neutralize *exactly* with an alkali and test with Fehling's solution. Does any reduction take place? Explain. Write out all reactions. Save a specimen of pure cellulose.

EXPERIMENT 42.

SCHWEIZER'S REAGENT.

Dissolve 10 grams of crystallized copper sulphate in 150 to 200 c.c. of water in a 500 c.c. flask, and add to the cold solution 5 c.c. of a solution of ammonium chloride. Precipitate cupric hydroxide from this solution by adding a slight excess of sodium hydroxide solution. Then wash by decantation with cold water, and finally on a cloth filter, until the wash water no longer gives a precipitate with barium chloride solution. To 30 c.c. of ammonia solution add this washed copper hydroxide as long as it will dissolve, and filter the solution through glass wool. This is an ammoniacal solution of copper hydroxide, and is known as Schweizer's Reagent. Will it dissolve cellulose? Try it with some ordinary cotton, with filter-paper, and with some pure cellulose. Dilute the solutions thus obtained with water, filter through glass wool (?), and acidify with dilute hydrochloric acid. What takes place?

It is essential for the success of the above experiment that the copper hydroxide should be washed free from salts and preferably out of contact with the air.

Explain the Willesden process of water-proofing cotton fabrics. (See Watt's Dictionary of Chemistry, revised edition, or Thorpe's Dictionary of Applied Chemistry, under *Cellulose*.)

EXPERIMENT 43.

STARCH.

Grind in a small mortar a few grains of arrowroot starch with some distilled water. Pour the creamy mass thus produced into 500 c.c. of boiling distilled water contained in a porcelain dish, stirring constantly while the starch is being added. A few drops of this solution is then added to a liter of water and *one drop* of a solution of potassium iodide. Does any color appear? Add a drop or two of freshly prepared (?) chlorine water and note what takes place. Explain. Is the color destroyed when chlorine water is added in excess? when alkalies are added? by sulphurous acid? by hydrogen sulphide? by sodium thiosulphate? Try it with each reagent with a small quantity in a test-tube. Explain and write out all reactions. Does the color disappear on heating? Heat some in a test-tube, and then cool. Does the color reappear on cooling? Explain. From the above conduct state whether the starch-iodine compound is a chemical compound or not. Test the delicacy of this reaction for iodine and for starch.

EXPERIMENT 44.

Study the action of bromine water on some of the starch solution made in Experiment 43. Does it give any color? Add a few drops of a solution of potassium bromide to some of the starch paste solution, dilute with water, and then add a few drops of chlorine water. What takes place? Why is the chlorine water added? Is the starch-bromine compound destroyed by heat? Try it. Is this reaction between the starch and bromine as delicate as the one with iodine? Could you distinguish between chlorine, bromine, and iodine by means of starch solution?

EXPERIMENT 45.

Test some of the starch paste solution made in Experiment 43 with Fehling's solution. Does it reduce it? Add 5 c.c. of concentrated hydrochloric acid to 200 c.c. of the starch paste solution, and heat to boiling in a flask connected with a return condenser for an hour and a half. When cold, neutralize with sodium carbonate solution, and examine with Fehling's solution. Does it reduce the Fehling's solution now? Does the solution now contain any starch? Test it with a solution of iodine in potassium iodide solution. Explain, and write out all reactions.

EXPERIMENT 46.

DIETHYL OXALATE AND OXAMIDE.

Grind 100 grams of crystallized oxalic acid to powder, and dehydrate by heating in an air-bath to 100° C. Weigh the *anhydrous* oxalic acid, and add to it an equal weight of absolute alcohol (97% will do). Boil this mixture in a flask connected with a return-condenser for four hours. Then transfer to a distilling-flask, and distil until the temperature reaches 110° C. Add to the residue left in the distilling-flask a volume of absolute alcohol equal to that of the distillate, and again boil for four hours in the apparatus first used. Once more transfer to the distilling-flask, and distil. When the temperature reaches 145° C., change the receiver, increase the heat, and distil off the residue *as quickly as possible.* From the first fractions separate the ethyl formate, and, from the second, the diethyl oxalate, by fractional distillation, using a Hempel distilling-tube.

Determine the boiling-point, color, taste, odor, specific gravity, inflammability, etc., of both substances, and explain their formation.

To the alcoholic solution of diethyl oxalate, resulting from the fractionation, add some concentrated ammonia. What takes place? Filter off the precipitate, wash, and examine. Boil some with a little caustic potash solution in a test-tube. Does it dis-

solve? What gas comes off? What remains in solution? Prove it.

Save specimens of ethyl formate, diethyl oxalate, and oxamide. Write out all reactions.

EXPERIMENT 47.

UREA FROM URINE.

(HOOD.)

Evaporate 3 liters of fresh urine to a thick syrup in a porcelain dish, first on a gas-stove, then on the water-bath. When *cold*, add concentrated nitric acid to the residue, and precipitate the urea contained in the syrup in the form of the nitrate. Filter off the precipitate, using a Witt plate and suction-pump. Dissolve the crude nitrate in boiling water, and decolorize the solution by adding a dilute solution of potassium permanganate, drop by drop. Then evaporate the solution to crystallization, filter, and continue the evaporation with the mother liquor. Drain, and dry the urea nitrate, and save a small specimen.

Weigh the nitrate, dissolve in water, add a slight excess of powdered barium carbonate (calculated), and evaporate to dryness on the water-bath. From the dry residue extract the urea by boiling with strong alcohol (95%), filter, and evaporate to crys-

tallization. Weigh the urea obtained, and calculate the percentage in the urine used. Determine crystal form, melting-point, solubility, color, taste, odor, etc. Heat a little of the dry substance. What is formed? Treat a concentrated solution in a test-tube with a solution of oxalic acid, and explain what takes place. Add a dilute solution of urea to a dilute solution of mercuric nitrate. What takes place? Add a little mercuric oxide to a solution of urea. What takes place?

Save a specimen of urea, and write out all reactions.

EXPERIMENT 48.

UREA FROM AMMONIUM CYANATE.

Dissolve 20 grams of the potassium cyanate obtained as directed in Experiment 24, page 31, in water, and add to it a solution of ammonium sulphate, containing just enough ammonium sulphate to change the potassium cyanate to potassium sulphate (calculated). Evaporate the solution to dryness on the water-bath, and extract the residue with boiling alcohol (99%). Filter and evaporate to crystallization. Weigh the urea obtained, and see how it agrees with the amount required by theory. Compare the artificial urea with that obtained from urine, and prove their identity by a determination of melting-points and other physical and chemical properties. What is formed when ammonium sulphocyanate is heated to its melting-point?

Save a specimen of the urea, and write all reactions.

EXPERIMENT 50.

Dissolve 8 grams of caustic soda in 100 c.c. water, and add 2 c.c. of bromine. What does this solution contain? Make a solution of both the artificial and the natural urea, and add to some of the above solution. A gas is given off. Collect some, and determine what it is. What remains in solution? Urea may be determined in urine by this method. Explain process, and write out reactions.

Dissolve a small quantity of sodium nitrite in water, and add to it an acidified solution of urea. What takes place? Explain. Reaction?

EXPERIMENT 51.

ETHYLENE AND ETHYLENE BROMIDE.

(HOOD.)

In a 3-liter balloon-flask put a mixture of 25 grams of alcohol and 150 grams of concentrated sulphuric acid. Heat to 160°–170°, and add gradually, through a separating-funnel, reaching below the surface of the liquid, a mixture of 1 part alcohol and 2 parts concentrated sulphuric acid. Pass the gas through two wash-bottles containing caustic soda solution (?). Collect some of the gas over water. Determine odor, taste, color, inflammability, solubility, etc. Mix 1 volume of the gas and 3 of oxygen in a stout cylinder, and apply a flame. What takes place? After enough gas has been collected for the above experiments, pass it into another wash-bottle containing a layer of bromine, $\frac{1}{4}$ inch deep, covered with water, and then into a bottle containing only water. When the bromine has been decolorized (explain), separate from the water, wash the resulting product with dilute caustic soda solution, then with water, separate from the water, dry with calcium chloride, and distil, noting the temperature.

Determine its boiling-point, color, taste, specific gravity, odor, solubility, etc. Does it solidify on cooling? What is formed when it is treated in alcoholic solution with granulated zinc? Prove it. Save specimen of ethylene bromide, and write out all reactions.

EXPERIMENT 52.

ACROLEIN.

(Hood.)

Heat in a small flask a mixture of 5 grams of *anhydrous* glycerol and 10 grams of powdered fused acid potassium sulphate. Pass the gases evolved through a bent tube into water contained in another small flask. Note the odor. What produces it? Does the aqueous solution reduce an ammoniacal solution of silver oxide? Try it. Explain. Save tube containing mirror, and write out all reactions.

EXPERIMENT 53.

ACETYLENE.

Screw a Bunsen burner into an iron or brass tube, bent at an angle of forty-five degrees, and connected with, first, an empty bottle, and then with a wash-bottle containing an ammoniacal solution of silver oxide,[1] and then with a suction-pump. Light the burner at the *base*, and start the pump. Regulate the air and gas so that the gas is slightly in excess (?), and a flame is obtained like that formed when a burner "strikes back." A precipitate is formed. What is it? After sufficient has collected, filter it off, wash with water, and examine. Determine color, solubility, etc. Does it resemble silver chloride in any respect? Explain. Does it explode when heated or on percussion? Dry some *carefully*, and try it. Write all reactions. Save silver solutions, and put into bottle marked "Silver Residues."

[1] To make ammoniacal silver oxide solution, proceed as follows: Dissolve 5 grams of silver nitrate in water, add cautiously dilute ammonia solution till the precipitated silver oxide is just dissolved. An ammoniacal solution of cuprous chloride may be substituted for the silver solution.

EXPERIMENT 54.

Put the precipitate obtained in Experiment 53, together with a little water, into a small flask provided with a funnel-tube and delivery-tube. Slowly add, through the funnel-tube, moderately concentrated nitric acid, and warm the flask gently if necessary. A gas is evolved. After the air has been expelled, collect the gas over water in cylinders, and examine. What is it? Has it any odor? Does it burn? Does it form an explosive mixture with oxygen or air? Try it. Does it unite with bromine? Try it with some bromine water. Is it soluble in water? Is it lighter or heavier than air? What remains in the flask from which the gas was evolved? Explain and write all reactions. Is acetylene an acid?

Save silver salts, and put into proper bottle.

EXPERIMENT 55.

BENZENE.

Grind together intimately in a mortar 50 grams of benzoic acid and 100 grams of good soda-lime. Distil the mixture from an iron retort, connecting the long delivery-tube of the retort with a bent adapter, and surrounding the receiver with ice-water. Heat the retort with a triple burner, and continue the heating until liquid ceases to distil. Separate the oil from the water in the distillate, wash with dilute caustic soda solution (?), dry with fused calcium chloride, as usual, and re-distil from a small distilling-flask, noting the temperature at which it boils. What is the substance thus obtained?

Determine its boiling-point, specific gravity (lighter or heavier than water), odor, color, taste, inflammability, etc. Does it solidify when cooled to 0°? Try it. Is it a good solvent? Try it. What remains in the retort in which this substance was made? Prove it. Is there any analogy between this method of making benzene and the method of making marsh gas? Save a specimen of the benzene, and write out all reactions.

EXPERIMENT 56.

BENZENE AND TOLUENE.

Put 800 c.c. of light oil into a 1-liter distilling-flask connected with a condenser and receiver. Distil until the temperature reaches 125°. Put the distillate into a large separating-funnel, and shake it several times with small quantities of concentrated sulphuric acid. Remove the acid, wash with water, and then shake once or twice with caustic soda solution. Remove the soda solution, wash again with water, dry with fused calcium chloride in the usual manner, and subject the product to fractional distillation, using a Hempel distilling-tube. Continue the fractionation until a portion is obtained boiling at about 80°, and another at about 110°. Prove that the first fraction is identical with the benzene obtained in Experiment 55. What is the second fraction? What is the object of the shaking with sulphuric acid and the caustic soda solution? Save specimens of both substances.

EXPERIMENT 58.[1]

NITROBENZENE.

(Hood.)

A cooled mixture of 60 grams of concentrated nitric acid (sp. gr. 1.42) and 80 grams of ordinary concentrated sulphuric acid (sp. gr. 1.84) is slowly and carefully added from a separating-funnel, drop by drop, and with constant shaking, to 50 grams of benzene, contained in a $\frac{1}{2}$-liter flask, care being taken to prevent the temperature rising above 50°. Gases come off. What are they? Explain. When all the acid has been added, heat the mixture on a boiling-water or steam bath for half an hour, shaking the flask constantly. After cooling, pour the mixture into half a liter of cold water, and separate the acid layer by means of a separating-funnel. Wash the oil remaining several times with water, and then with dilute soda solution, drawing off the oil each time from the *bottom* of the funnel. Wash finally with water, separate the oil from the water as completely as possible, dry with calcium chloride in the usual way, and subject to distillation, using a plain tube as a condenser. What comes over? What is the brownish residue left in the flask? Determine the boiling-point, specific gravity, solubility, color, taste, odor, etc., and yield of the nitro-

[1] The vapor of nitrobenzene is poisonous, and care should be taken not to inhale it.

benzene obtained. Does it solidify on cooling? Why is the sulphuric acid added in the preparation of the nitrobenzene?

Write out all reactions, save a small specimen of the substance, and use the rest in the following experiments.

EXPERIMENT 59.[1]

DINITROBENZENE.

(HOOD.)

1.

Add gradually 1 volume of benzene (50 grams) to 2 volumes of fuming nitric acid (sp. gr. 1.52), warming the mixture toward the end of the operation until all the benzene has gone into solution. Allow the mixture to cool somewhat, then add 3.3 volumes of concentrated sulphuric acid, and boil for some time. After cooling, pour the mixture slowly into water. Filter off the crystal mass on a Witt plate, wash with water until free from acid, drain completely, and recrystallize from hot alcohol.

Determine the melting-point, crystal form, solubility, color, odor, taste, etc. What substances remain in the mother liquor? Save a specimen of the crystals, and write out all reactions.

[1] Make dinitrobenzene either by this method or by the one given in the experiment following.

EXPERIMENT 59.

DINITROBENZENE.

(HOOD.)

2.

15 grams of nitrobenzene are gradually added from a separating-funnel to a mixture of 25 grams of concentrated nitric acid (sp. gr. 1.47) and 40 grams of concentrated sulphuric acid (sp. gr. 1.84), contained in a flask of 500 c.c. capacity. The flask should not be cooled, but should be frequently shaken during the addition of the nitrobenzene. When all the nitrobenzene has been added, heat the flask gently on a sand-bath until red fumes cease to be given off and the reaction is complete. The flask should be shaken constantly during the heating. After cooling, pour into a liter of water. Filter off the crystals on a Witt plate, using a suction-pump to drain thoroughly, wash free from acid, drain, and recrystallize from hot alcohol.

Determine the melting-point, crystal form, solubility, color, odor, taste, etc., of the crystals. What substances remain in the alcoholic mother liquor? Save a specimen of the crystals, and write out all reactions.

EXPERIMENT 60.

ANILINE.

Put 80 grams of water, 55 grams of finely divided cast-iron filings,[1] and 50 grams of nitrobenzene into a 1500 c.c. round-bottomed flask connected with a return-condenser. Add 10 grams of concentrated hydrochloric acid through the inner tube of the condenser. Heat gently with a small flame until the reaction begins. After the first violent reaction is over, heat the flask, and continue the heating until the odor of nitrobenzene has disappeared. Then add water (500 c.c.) to the flask, and distil with steam. *If* the distillate has the odor of nitrobenzene, add conc. hydrochloric acid until the aniline has entirely dissolved, and extract the nitrobenzene by shaking with ether. Then remove the ether, concentrate the aqueous solution, and add caustic soda solution to alkaline reaction, put into a separating-funnel, and extract two or three times with small quantities of ether. If the distillate does not smell of nitrobenzene, and a little of the oil dissolves *completely* in hydrochloric acid, simply extract it once or twice with ether. Separate the ether extracts from the water as completely as possible, dry with solid caustic potash in the usual manner, place in a distilling-flask, and distil off the ether. Finally, increase the heat, and distil the aniline. Determine

[1] Ferrum met. subt. pulv. of Schuchardt.

its boiling-point, solubility, specific gravity (lighter or heavier than water), color, odor, taste, etc. Has the solution in water an alkaline reaction? Try it. Does aniline dissolve in dilute acids? Explain. Is it affected by the light? Does its aqueous solution precipitate salts of zinc, aluminum, and iron? Try it. Explain. Does aniline decompose ammonium salts? Heat some with a solution of ammonium chloride.

Save a small specimen of aniline, and use the rest in Experiments 61 and 62. Write out all reactions. (See Roscoe and Schorlemmer's Treatise on Chemistry, Vol. III. page 196, for explanation of the process.)

EXPERIMENT 61.

To a dilute aqueous solution of some of the aniline made in Experiment 60 add a few drops of a filtered solution of bleaching-powder, and note the result. Explain. *Very* dilute solutions of aniline give but a slight coloration, but a color is brought out by adding a few drops of a dilute solution of ammonium sulphide to the mixture. Try it, and test the delicacy of this last reaction.

To a solution of aniline in concentrated sulphuric acid add a few grains of solid potassium bichromate, and warm the test-tube gently. What takes place?

What other reactions have you already had which might be used to detect the presence of aniline? Write out all reactions after the aniline dyes have been considered.

EXPERIMENT 62.

DIAZOBENZENE SULPHATE.

Dissolve 15 grams of the aniline obtained in Experiment 60 in 9 to 10 times its weight of alcohol (95%), and add cautiously 25 grams of concentrated sulphuric acid, at first slowly, afterwards more rapidly. What is formed? Cool the mixture to 15° C., add 20 grams of amyl nitrite,[1] and place the flask in a freezing mixture, shaking the flask constantly until crystallization begins. Filter off the crystals on a Witt plate, drain thoroughly by suction, wash with alcohol (small quantity) and then with ether, and dry in the air on filter-paper. By adding to the filtrate half its volume of ether, and again placing in the freezing mixture, a further quantity of the salt may be obtained.

Determine color, solubility, crystal form, etc., and yield of the salt. Does the dry salt decompose in the light?

(*a*) Heat some of the dry salt *cautiously* on platinum foil, and note what takes place. Does the salt explode on percussion? Try it.

(*b*) Boil some with absolute alcohol, and describe what takes place. What are the products of the reaction?

(*c*) Boil a little of the dry salt with water. What

[1] Do not breathe the vapor of amyl nitrite (?).

takes place? What are the products of the reaction? Prove it.

(*d*) Dissolve the rest of the dry diazo salt in cold water, and add slowly the theoretical amount of potassium iodide in water. What takes place?

Distil off the oil formed with steam, separate from the water in the distillate, wash with caustic soda solution, dry with calcium chloride, and distil, noting the temperature. What is the substance thus obtained? Determine the color, odor, action of light, solubility, boiling-point, and specific gravity (lighter or heavier than water) of the substance. Save a specimen. Write out all reactions.

EXPERIMENT 63.

BENZENESULPHONIC ACID.

Boil gently for about thirty hours, on a sand-bath, a mixture of equal volumes of benzene (100 grams) and concentrated sulphuric acid (200 grams) in a 500 c.c. round-bottomed flask connected with a return-condenser, until all the benzene is used up. What is formed? When cold, pour the contents of the flask gradually into three or four liters of water contained in a large evaporating-dish. Heat the dish gently on a gas-stove under the hood, and add little by little, with constant stirring, powdered chalk or slaked lime, until the acid is neutralized. Filter the hot solution through muslin, or by the method of reverse filtration (see Experiment 9), wash the residue thoroughly with hot water, and evaporate the combined filtrates to a small volume (500 c.c.), filtering off the gypsum which separates out from time to time. Add carefully a saturated solution of potassium carbonate until all the calcium is precipitated. Filter, wash, dry, and save the calcium carbonate. Evaporate the combined filtrates to dryness, first heating it on the gas-stove, and then on the water or steam bath. Powder and heat, in a small porcelain dish, to 100°–120° in an air-bath. Crystallize some from alcohol, save a small specimen of the crystallized potassium salt, and determine color, crystal form, etc. Use the dry salt in following experiments, and write out all reactions.

EXPERIMENT 64.

BENZENE SULPHON CHLORIDE AND SULPHONAMIDE.

(Hood.)

Grind together, in a porcelain dish, equivalent quantities (20 grams) of dry potassium benzene-sulphonate and phosphorus pentachloride, and heat the mixture on the water-bath until all the phosphorus oxychloride has been driven off. What is formed? Then add water, and wash the oil free from inorganic salts. Separate as completely as possible from the water, add 100 c.c. of *concentrated* ammonia solution, with constant stirring, and evaporate to dryness on the steam-bath. What is formed? Recrystallize the product from hot water, and determine its melting-point, crystal form, solubility, color, taste, odor, etc. Does the substance dissolve more readily in ammonia solution than in water? Explain. Write out carefully all reactions, and explain each step. Save specimens of the substances made.

EXPERIMENT 65.

PHENYL CYANIDE (BENZONITRILE).

Grind together intimately 40 grams of dry potassium benzene-sulphonate and an equal weight of potassium cyanide, and distil the mixture from an iron or copper retort, the delivery-tube of which is connected with a receiver by means of a bent adapter. What substance distils? Has it a bad odor (cf. Experiment 30)? What remains in the retort? Prove it. Put the oil into a 500 c.c. flask, add 150 c.c. of strong caustic potash solution, connect the flask with a return-condenser, and boil the contents until the oil disappears. What is formed? What gas is given off? After cooling, acidify the solution with hydrochloric acid. Filter off the precipitate formed, and recrystallize it from hot water. Determine its melting-point and properties. What is it? Write out all reactions carefully, explaining each step, and save a specimen of the last substance obtained.

EXPERIMENT 66.

PHENOL.

Dissolve 20 grams of the dry potassium benzene-sulphonate in as small a quantity of hot water as possible in a silver, nickel, or iron crucible, and add 35 grams of solid caustic potash. Heat the crucible in an oil-bath or Meyer air-bath (see Lassar Cohn, page 200), or with a direct flame, to 250°–252° C., for an hour, with constant stirring. The temperature is taken with a thermometer placed in a thin glass or copper tube closed at one end. When cold, dissolve the melted mass in a small quantity of hot water, acidify with hydrochloric acid (what gas is given off?), and take up the oil, which separates, with ether. Separate the ether extract, dehydrate it with potassium carbonate in the usual way, put it into a distilling-flask, connected with a condenser and receiver, and evaporate off the ether. Then increase the heat and distil the oil, which should be afterwards cooled in a freezing mixture, and the crystals drained thoroughly on a Witt plate by suction. Determine odor, taste (poison), color, crystal form, boiling-point, solubility, etc. By what other method have you already made this substance? Make a solution in water, and perform the following experiments: —

1. Add a few drops of ferric chloride solution.
2. Add $\frac{1}{4}$ volume of ammonia solution, and then

a few drops of a solution of bleaching-powder (1 part to 20 of water).

3. Add a few drops of bromine water.

Describe what takes place in each case, and write out all reactions. Save a specimen of the substance.

EXPERIMENT 67.

ORTHO AND PARANITROPHENOL.

(Hood.)

Mix 80 grams of nitric acid (sp. gr. 1.34) and 120 grams of water in a $\frac{1}{2}$-liter flask. Cool the mixture with cold water, and add to it, gradually, and with constant shaking, 40 grams of melted phenol. After the mixture has stood twelve hours, pour off the acid liquid from the brown oil formed, wash the oil once or twice with water to free it from acid, and after neutralizing the combined acid liquid and wash-water with sodium carbonate, subject the brown oil and wash-liquors to distillation in superheated steam. For this purpose generate the steam in a copper boiler and pass it through a copper coil, heated by means of a triple burner, into a round-bottomed copper or glass flask of about 3 liters' capacity, containing the oil and wash-liquors, and which is heated by means of a burner (see Lassar-Cohn, page 17). What passes over with the steam? Continue the distillation until the distillate passes over almost colorless, and the residue in the distilling-flask no longer has the odor of the volatile oil. Filter off the solid in the receiver, drain, and recrystallize from alcohol.

After the distilling-flask has cooled, filter off the crystals and extract the black resinous mass *repeatedly* with boiling hot concentrated hydrochloric acid.

Decant off the hydrochloric acid solution from the tarry mass, concentrate by evaporation, and allow to cool. What are the crystals which separate? Recrystallize them from the same solvent, if colored. Determine the crystal form, melting-point, solubility, volatility, color, odor, taste, etc., and yield of both substances. Have they acid properties? Explain. Write out all reactions, and save specimens of the two nitro-phenols.

EXPERIMENT 68.

PICRIC ACID.

(HOOD.)

Heat together in a round-bottomed $\frac{1}{2}$-liter flask to 100° C. a mixture of 20 grams of phenol and 20 grams of concentrated sulphuric acid until *complete* solution takes place. What is formed? Remove the burner, dilute with twice the volume of water, and add the solution (from a separating-funnel, and with constant shaking), gradually and *carefully*, to 100 grams of nitric acid (sp. gr. 1.4). Warm the mixture on the water-bath until the red color changes to yellow, then pour into a liter of water, filter off the crystals on a Witt plate, drain thoroughly, wash with water, and purify by recrystallization from hot water containing .1% sulphuric acid. Saturate some of the crude picric acid with a solution of sodium carbonate, and add to the hot filtered solution a few crystals of sodium carbonate. What takes place? Explain. Determine color, odor, taste (poison), crystal form, melting-point, solubility, etc., of the acid. Does the solution of the acid dye silk or wool? Does the dry sodium salt or the acid explode when heated or struck with a hammer? Save specimens of both the acid and the sodium salt, and write out all reactions.

EXPERIMENT 74.

PHTHALIC ACID.

(Hood.)

Grind to a fine powder 40 grams of naphthalene, and then 80 grams of potassium chlorate, mix and grind together. Add just enough water to form a thick paste, so that the mass may be rolled up into small balls. Dry these at ordinary temperature, and then add them slowly, one by one, to 400 grams of concentrated hydrochloric acid, contained in a 1-liter flask. The flask should not be allowed to get hot, and should be shaken constantly. What is formed? Pour off the acid solution from the pasty mass, and wash with lukewarm water once or twice, and then digest several times with alcohol; pour off the alcohol completely, and dry at 80°. Put the purified substance thus obtained into a retort with upright neck, and boil with ten times its weight of nitric acid (sp. gr. 1.45). The nitric acid should be added a little at a time, and, after boiling awhile, more is to be added, and so on. When the oil has passed completely into solution distil off the nitric acid, and after changing the receiver, sublime the residue in a current of air. What passes over? Save a specimen of this substance, and recrystallize the rest from water. Determine its melting-point, crystal form, color, taste, etc. Is it an acid? Save a specimen of the acid, and write out all reactions.

EXPERIMENT 75.

SALICYLIC ACID.

Boil 10 c.c. of oil of wintergreen with 100 c.c. of a 20% solution of potassium hydroxide in a round-bottomed flask connected with a return-condenser until it has all disappeared. What is formed? When cool, acidify with hydrochloric acid. Filter off the precipitated substance, recrystallize it from water, and determine its melting-point, color, crystal form, taste, etc. What change takes place when some of the substance is carefully heated in a dry test-tube? What are the products? Prove it. Dissolve some of the acid in water, and add a few drops of a solution of ferric chloride. What takes place? Write out all reactions, and save a specimen of the acid.

EXPERIMENT 76.

SALICYLIC ACID.

Dissolve 50 grams of phenol in the equivalent quantity of a solution of sodium hydroxide (22 grams $NaOH$ to 100 c.c. H_2O), and evaporate in a flat iron dish with a direct flame, stirring constantly when the mass becomes pasty, and lowering the flame. When dry enough, powder, and continue the heating until *all* the moisture is driven off. What is this substance? Put the dry substance into a small tubulated retort connected with a receiver, and heat the retort in an oil or metal bath (or air-bath) until the temperature within the retort is 100°, then pass in *dry* carbon dioxide. Let the temperature in the retort rise gradually until in the course of two hours it has reached 180°. What substance distils? Finally, let the temperature rise to 220°–250°. When nothing more distils at this temperature stop the operation. What remains in the retort? Dissolve the contents of the retort in a little water, and acidify with hydrochloric acid. What takes place? Filter off the crystals on a Witt plate, wash with water, and recrystallize from hot water, decolorizing the solution with boneblack if necessary. Compare the substance thus made with that obtained in Experiment 75. Are the two identical? Prove it. Save a specimen of the acid, write out all reactions, and explain the process.

EXPERIMENT 77.

SALICYLIC AND PAROXYBENZOIC ALDEHYDES.

Dissolve 50 grams of phenol in a solution of sodium hydroxide (containing 100 grams of sodium hydroxide to 170 grams of water) in a 1-liter round-bottomed flask. Connect with a return-condenser, heat the flask in a water-bath until the temperature of the bath reaches 60°, and then add gradually through the inner tube of the condenser, with thorough shaking, 75 grams of chloroform. Complete the reaction by heating the contents of the flask to boiling for an hour or two. Then distil off the unused chloroform with steam. Acidify the contents of the flask with dilute sulphuric acid and again distil in steam. What distils with the water? When drops of oil cease to distil, extract the distillate with ether once or twice, and shake the *concentrated* ethereal solution in a separating funnel with a *saturated* solution of sodium acid sulphite.[1] What takes place? Explain. Draw off the lower layer, filter, and shake the filtrate again with the ethereal solution, and again filter. Continue this process as long as crystals form. Wash the crystals on a Witt plate with alcohol, drain completely by suction, and decom-

[1] The sodium bisulphite solution should be freshly made, and is best prepared by placing powdered sodium carbonate in a flask, covering it with a layer of water and saturating with sulphur dioxide. The commercial sodium bisulphite will not answer for the purpose.

pose them by warming with an excess of dilute sulphuric acid. When cold, take up the oil resulting with ether, dehydrate the ethereal solution with calcium chloride, put into a distilling-flask, evaporate off the ether, and finally increase the heat, and distil the substance. Determine odor, color, boiling-point, specific gravity, solubility, volatility, etc., of the substance, and save specimens of it and its sodium bisulphite compound.

Filter the contents of the flask (from which the oil was distilled), while *hot*, through a wet filter, and extract the cold filtrate with ether. Evaporate off the ether, and recrystallize the product from hot water. What is it? Determine color, crystal form, melting-point, solubility, volatility, etc. Show how you could distinguish between the two aldehydes by the reaction with ferric chloride. Save a specimen of the substance, and write out all reactions.

EXPERIMENT 78.

ROSANILINE.

Heat 1 gram of a mixture of aniline and para-toluidine (2 molecules of aniline and 1 of paratoluidine) in a test-tube or small flask in an oil or sulphuric acid bath (Hood) for one and a half hours to 180°–200° C., together with 3 grams of mercuric chloride and 2 grams of aniline. What is formed? Extract the melted mass with alcohol, filter, and evaporate to dryness on the water-bath. Does the aqueous solution of the substance dye wool, silk, or cotton? Try it. Save a specimen of the colored solution, and write out the reaction.

EXPERIMENT 79.

Dissolve 2 or 3 grams of picric acid in *hot* water containing a small amount of sulphuric acid. Steep in this hot solution a piece of white yarn or flannel and a piece of white silk. Press out the excess of solution, and dry; then do the same thing with a piece of white cotton or linen cloth, and dry. Add to a solution of lead acetate some alum solution; soak a piece of white cotton or linen cloth in this solution, and dry partially. Then steep in the picric acid solution, press out the excess of the solution, and dry. Determine which of the materials are dyed permanently by washing them all in hot water and drying. Explain. Save samples of the dyed material.

EXPERIMENT 80.

INDIGO WHITE.

Dissolve some stannous chloride (tin salts) in alcohol, and add enough of an alcoholic caustic soda solution to dissolve the stannous acid precipitated, and then a slight excess of the alkali. Add this solution to some powdered indigo in a flask, and warm the solution gently. Decant the clear solution, and pass a current of air through it, or expose it to the air in a shallow dish. What takes place? Explain the whole process.

EXPERIMENT 81.

ANTHRAQUINONE.

Dissolve 25 grams of pure anthracene in the necessary quantity of boiling glacial acetic acid, contained in a round-bottomed flask connected with a return-condenser. Filter the solution if necessary. Add gradually and in small portions, through the inner tube of the condenser, keeping the solution boiling, 45 grams of chromic acid dissolved in glacial acetic acid. Continue the heating until the solution becomes intensely green colored, then distil off the greater part of the glacial acetic acid and save it. Pour the residue into water. What is precipitated? After allowing to stand for awhile, filter, wash with water, hot dilute caustic soda solution, and finally with hot water. Recrystallize from hot glacial acetic acid or benzene. Determine color, crystal form, melting-point, solubility, etc., of the substance. Does it sublime? Warm one part of the substance with zinc-dust (2 parts) and caustic soda solution (30 parts of 50% solution). What takes place? Explain. Save a specimen of the anthraquinone, and write out all reactions.

EXPERIMENT 82.

ALIZARIN.

(Hood.)

Heat 20 grams of anthraquinone with 60 grams of concentrated sulphuric acid to 250°–260°, until, on taking out a small quantity and adding it to water, no anthraquinone is precipitated. What is formed? Pour the mixture cautiously into a large quantity of water contained in a porcelain dish, stirring the water vigorously. Warm, and neutralize while hot with chalk or slaked lime; filter through muslin or by the method of reverse filtration. Evaporate to about half the volume, filtering if necessary. Then add a saturated solution of sodium carbonate until all the calcium has been precipitated. Filter and evaporate to dryness, first over a free flame and then on the water or steam-bath. Save a specimen of the crystallized sodium salt, and convert the rest into alizarin. For this purpose dissolve one part of the sodium salt in as small a quantity of hot water as possible in a silver or nickel dish fitted into a copper bath (see Lassar-Cohn, page 200), add 5 parts of solid caustic soda and 0.3 parts of potassium chlorate. Heat the mixture, with *constant stirring*, for several hours to the temperature of boiling naphthalene. Let cool, and remove the melted mass by boiling with water and by mechanical means. Saturate the solution thus obtained with dilute sulphuric acid, boil for

fifteen minutes, filter while hot, and wash the precipitate with hot water until the sulphuric acid is removed. Dry and sublime. What is this substance? Determine its color, melting-point, crystal form, solubility, volatility, etc. Does it dissolve in alkalies? Explain. What is formed by heating some with ten times its weight of zinc-dust in a test-tube? Save a specimen of the alizarin, and write out all reactions.

R. D. Olson
Arcata, California.
Oct. 1906

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